Carderock Division
Naval Surface Warfare Center
Bethesda, Md. 20084–5000

CARDEROCKDIV–SSM–61–93/04 March 1993
Survivability, Structures, and Materials Directorate
Technical Report

Properties of Superconducting $YBa_2Cu_3O_{6+x}$ Ceramic Materials: Effect of Processing Parameters

by
A. Srinivasa Rao

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ABSTRACT

The effect of the sintering temperature, the sintering time and the addition of 5 wt.% silver on the average particle size, bulk density and the superconducting zero resistance temperature (T_c) and the normal state resistivity measured at 100 K of $YBa_2Cu_3O_{6+x}$ ceramic materials was investigated. The sintered specimens were produced from sintering the dry pressed powders (a) at different temperatures in the range 880 - 940°C for 2 hours and (b) at 920°C for different times in the range 0 - 10 hours. The results suggest that while the density and particle size of the sintered ceramic increases with an increase of the sintering temperature (and 2 hours of sintering), prolonged sintering (at 920°C) above 4 hours of duration does not improve the above properties. The normal state resistivity, measured at 100 K, decreases with an increase of both the sintering temperature and sintering time. However, the superconducting zero resistance temperature (T_c) does not change significantly. In the presence of 5 wt.% silver, it appears that both sintering temperature and duration of sintering significantly improves the bulk density, particle size and T_c of $YBa_2Cu_3O_{6+x}$. In addition, it also appears that if the sintering temperature is above 920°C, some $YBa_2Cu_3O_{6+x}$ particles tend to align in a preferred direction.

ADMINISTRATIVE INFORMATION

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INTRODUCTION

The electrical systems that utilize superconducting materials are presently used with liquid helium cryogenic system. Ceramic superconductors that have been identified in recent years can superconduct at liquid nitrogen temperatures (77 K) and at somewhat higher temperatures [1,2]. However, the new superconducting ceramic materials are very brittle and as such there are many difficulties in forming them into wires or other components. It has been suggested that the superplastic deformation can enhance the ease of fabrication of brittle ceramic materials [3]. The key to achieving superplastic deformation is to produce a ceramic preform with small grain size (typically 100 - 200 nm) and to subject the preform to a relatively constant stress at high temperature [4,5]. The small grain size in the preform translates into a large proportion of grain boundaries and it is the grain boundaries which slide under constant stress at high temperature and result in the superplastic deformation. The overall object of this project was to determine the set of sintering conditions, critical load and strain rates and the set of additives to maintain the required grain size during sintering and deformation. The aim of the present study is to understand the behavior of $YBa_2Cu_3O_{6+x}$ subjected to different sintering conditions (viz. temperature and time) with and without silver as an additive. In this paper, some of the experimental results relating the particle size, sintered compact density and the electrical properties (such as superconducting transition temperature, T_c, and the normal state electrical resistivity measured at 100 K) are presented.

EXPERIMENTAL

Figure 1 shows the typical flow diagram of the processing of $YBa_2Cu_3O_{6+x}$. The basic superconducting $YBa_2Cu_3O_{6+x}$ was synthesized from Y_2O_3, $BaCO_3$ and CuO using the solid state chemical reaction method. An aqueous suspension of the oxides of yttrium, barium and copper were mixed in a ball mill using zirconia balls for one hour. The suspension was dried in an oven maintained at 110°C for 24 hours. The dry powder was calcined in air at 940 $\pm$ 2°C for 6 hours and then slow cooled (typical rate of cooling 2°C per minute) to room temperature. The resulting material was then ground with a mortar and pestle.

The as-synthesized powder was divided into two equal portions. To one portion of the as-synthesized $YBa_2Cu_3O_{6+x}$ powder, 5 wt.% commercial silver powder (average size 1 micron) was added. The $YBa_2Cu_3O_{6+x}$/silver mixture was ball milled in dry powder state using zirconia balls for 30 minutes. Both powders (pure $YBa_2Cu_3O_{6+x}$ and silver/$YBa_2Cu_3O_{6+x}$) were dry pressed to produce a number of 2.54 cm diameter discs. Two different sets of sintered specimens were produced from the dry pressed discs. In the first set of specimens, the dry pressed discs were heated in a furnace at the rate of 10° C per minute in flowing oxygen till the sample temperature reached the required sintering temperature in the temperature range of 880 - 940°C. Once, the sample temperature has reached the required sintering temperature, the samples were maintained at that temperature for 2 hours. Later they were cooled very rapidly at the rate of 10°Cper minute. In the second set of experiments the dry

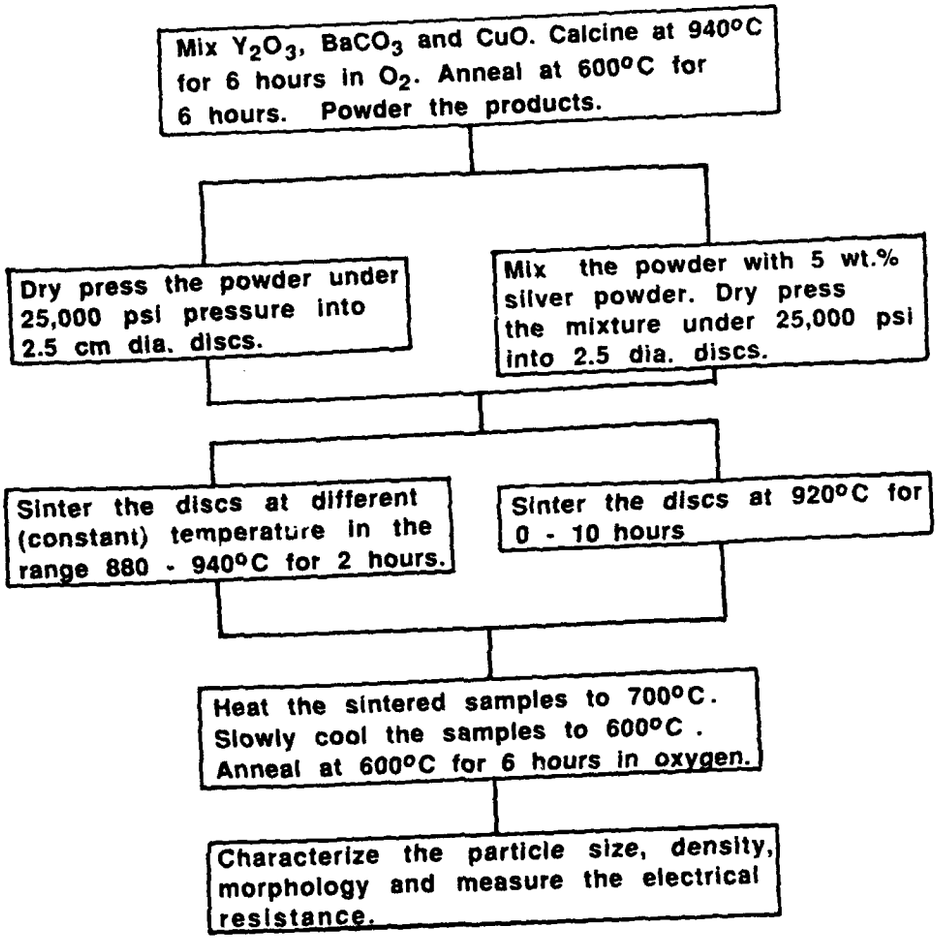

Figure 1. The flow diagram of the processing of $YBa_2Cu_3O_{6+x}$ superconducting ceramic materials with and without 5 wt.% silver.

pressed discs were rapidly heated to 920°C and were kept at that temperature (i.e. 920°C) for 0 - 10 hours. Afterwards the samples were cooled very rapidly at the rate of 10°C per minute. In order to obtain the required superconducting orthorhombic crystal phase, all samples were once again heated in oxygen to 650 - 700 °C at the rate of 10°C per minute. The samples were then cooled to 600°C at the rate of a2°C per minute and were held at that temperature for 6 hours before continuing a slow cool to the room temperature.

The silver was chosen an an additive, because the free silver will be plastic during deformation and may assist the onset of superplasticity. In addition, silver has been reported to improve the electrical properties of $YBa_2Cu_3O_{6+x}$ grain boundaries.

The particle size and surface area of the as-synthesized $YBa_2Cu_3O_{6+x}$ powder was determined using *sedigraph and single point* BET apparatus respectively. The particle size of sintered composites was estimated from a number of scanning electron micrographs obtained from sintered disc samples. The electrical resistance of all the samples was determined using four probe electrical resistivity meter. Indium solder was used to attach voltage and current leads to the samples and the applied current was in the range 10 - 100 milliamps. A nano voltmeter with a sensitivity of 5×10^{-7} volts was used. In order to remove the effects of thermally induced voltages from the resistance readings, the sign of the current was changed at each reading and the absolute value of the difference of the voltage readings was averaged and used in the plots of resistance versus temperature.

RESULTS

The as-synthesized $YBa_2Cu_3O_{6+x}$ has a density of ~ 5.9 gm cm^{-3} and a surface area of ~ 0.22 m^2gm^{-1}. The powder is poly dispersed and has an average particle size of ~ 10 microns. The density of the powder is comparable to the density values reported in the literature [6]. The $YBa_2Cu_3O_{6+x}$ particles tends to sinter into long elongated rods and retain primarily the orthorhombic crystal structure of $YBa_2Cu_3O_{6+x}$. The detailed analysis of both the morphology and crystal structure of these rods was given elsewhere [7].

The average particle size of $YBa_2Cu_3O_{6+x}$ resulted from sintering at a fixed sintering temperature in the range 880 - 920°C for 2 hours is shown in Figure 2(A). The sintered compact density corresponding to the above set of samples is shown in Figure 2(B). The results suggest that the particle size increases with an increase in the sintering temperature in the range 880 - 920°C. Above 920°C, the particle size remains independent of the sintering temperature. Although, the bulk compact density appears to increase with temperature, the gain in the density is not very significant. Figure 3 shows typical surface morphology of the $YBa_2Cu_3O_{6+x}$ particles as a function of sintering temperature for samples sintered at a constant temperature for 2 hours. The results suggests that the coarseness of the surface morphology of $YBa_2Cu_3O_{6+x}$ tends to decrease due to an increase in the sintering temperature. The results also suggests that partial melting of the $YBa_2Cu_3O_{6+x}$ occurs during sintering above 920°C for 2 hours. The

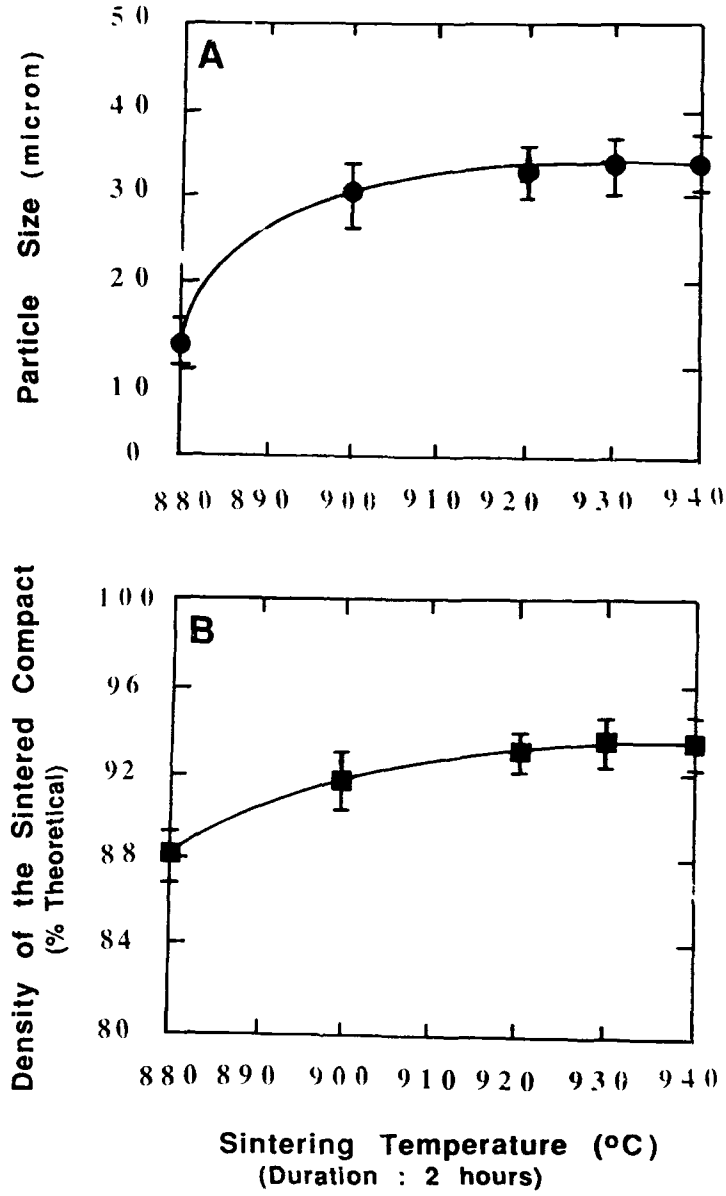

Figure 2. (A) The average particle size of $YBa_2Cu_3O_{6+x}$ and (B) the density of sintered compacts represented as a function of sintering temperature of pure $YBa_2Cu_3O_{6+x}$ ceramic materials. Sintering time : 2 hours.

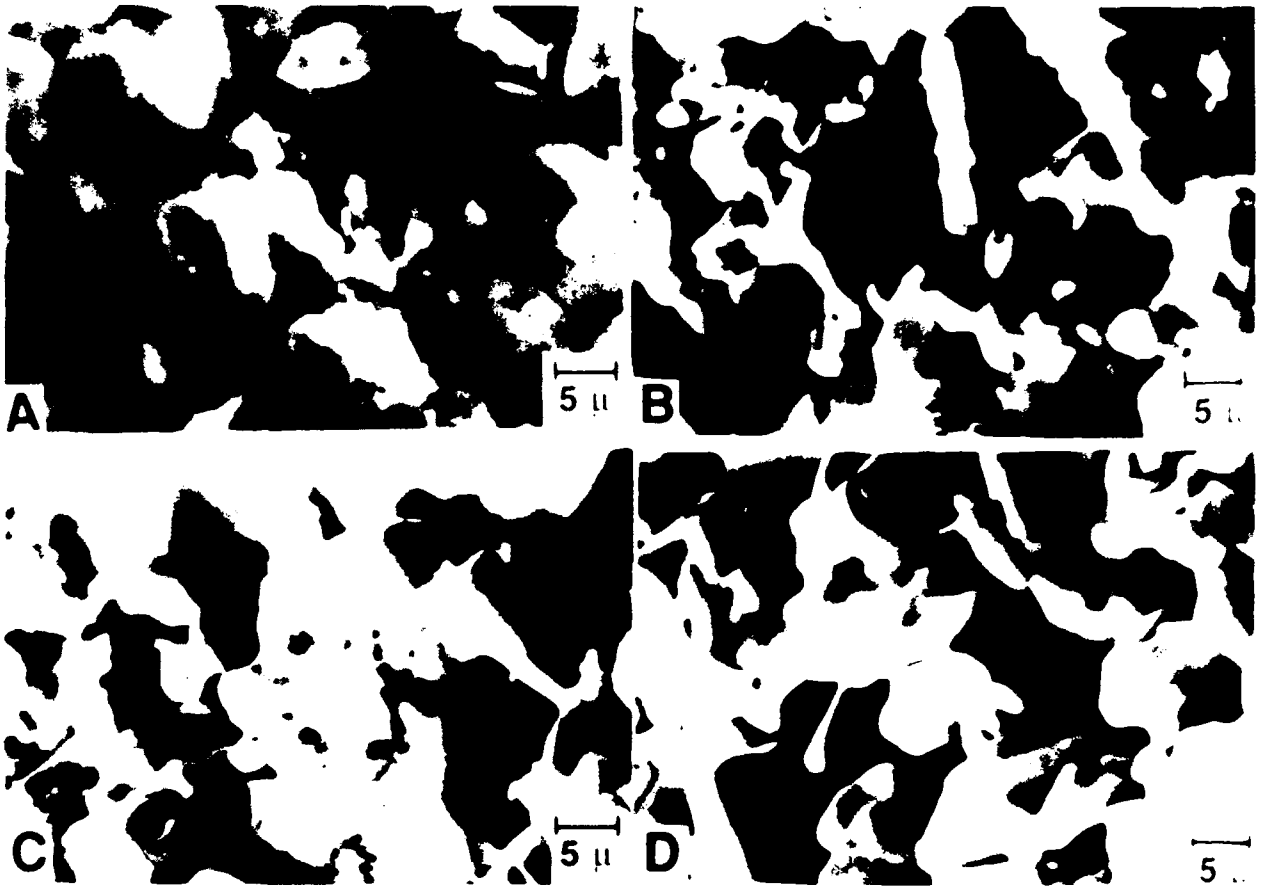

Figure 3. Typical morphology of $YBa_2Cu_3O_{6+x}$ ceramic materials sintered at (A) 900, (B) 920, (C) 930 and (D) 940°C for 2 hours.

results also indicate that the melting increases with an increase in the sintering temperature. Figure 4 shows typical electrical resistance versus temperature plots of sintered $YBa_2Cu_3O_{6+x}$ samples as a function of sintering temperature (sintering time of 2 hours). From such plots the zero resistance temperature (T_c) and the normal state resistivity at 100 K was determined and the results are shown in Figure 5. The results shown in Figures 4 and 5 suggest that while the zero resistance temperature increases with an increase in the sintering temperature, the resistivity of the samples decrease with an increase in over the sintering temperature.

Figure 6(A) and (B) shows the particle size and sintered compact density of $YBa_2Cu_3O_{6+x}$ ceramic materials sintered at 920°C for 0 - 10 hours. The results suggest that most of the particle growth due to sintering at 920°C occurs during the first 2 hours of sintering and prolonged calcination beyond 2 hours has very little effect on the particle size. The results on compact density indicates that the density of the sintered ceramic increases with an increase in the duration of sintering during the first 4 hours. Above the optimum sintering time of 4 hours, the continued calcination has no effect on the bulk density. Typical morphology of sintered $YBa_2Cu_3O_{6+x}$ particles is shown in Figure 7 as a function of sintering time at 920°C. The results suggest that the superconducting ceramic particles tend to lose the surface coarseness and exhibit smooth surface topology as a result of sintering at 920°C for 2 hours. However, an increase in the sintering time beyond 2 hours tends to melt the $YBa_2Cu_3O_{6+x}$ particles. Prolonged sintering at 920°C for more than 8 hours would

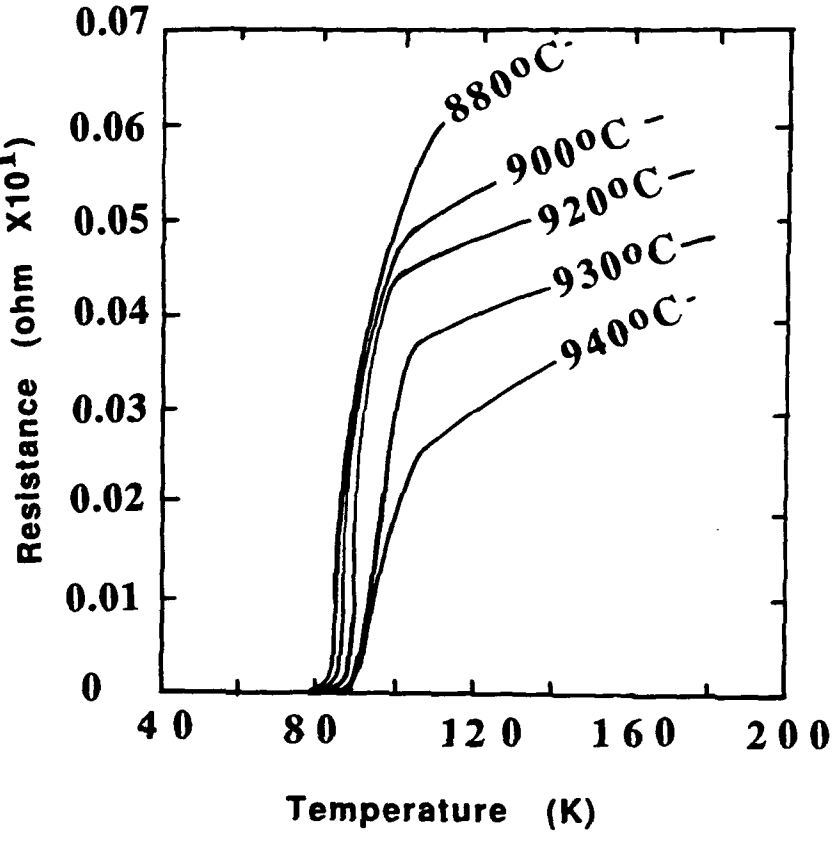

Figure 4. Electrical resistance versus temperature plots of pure $YBa_2Cu_3O_{6+x}$ ceramic materials sintered different temperatures for 2 hours.

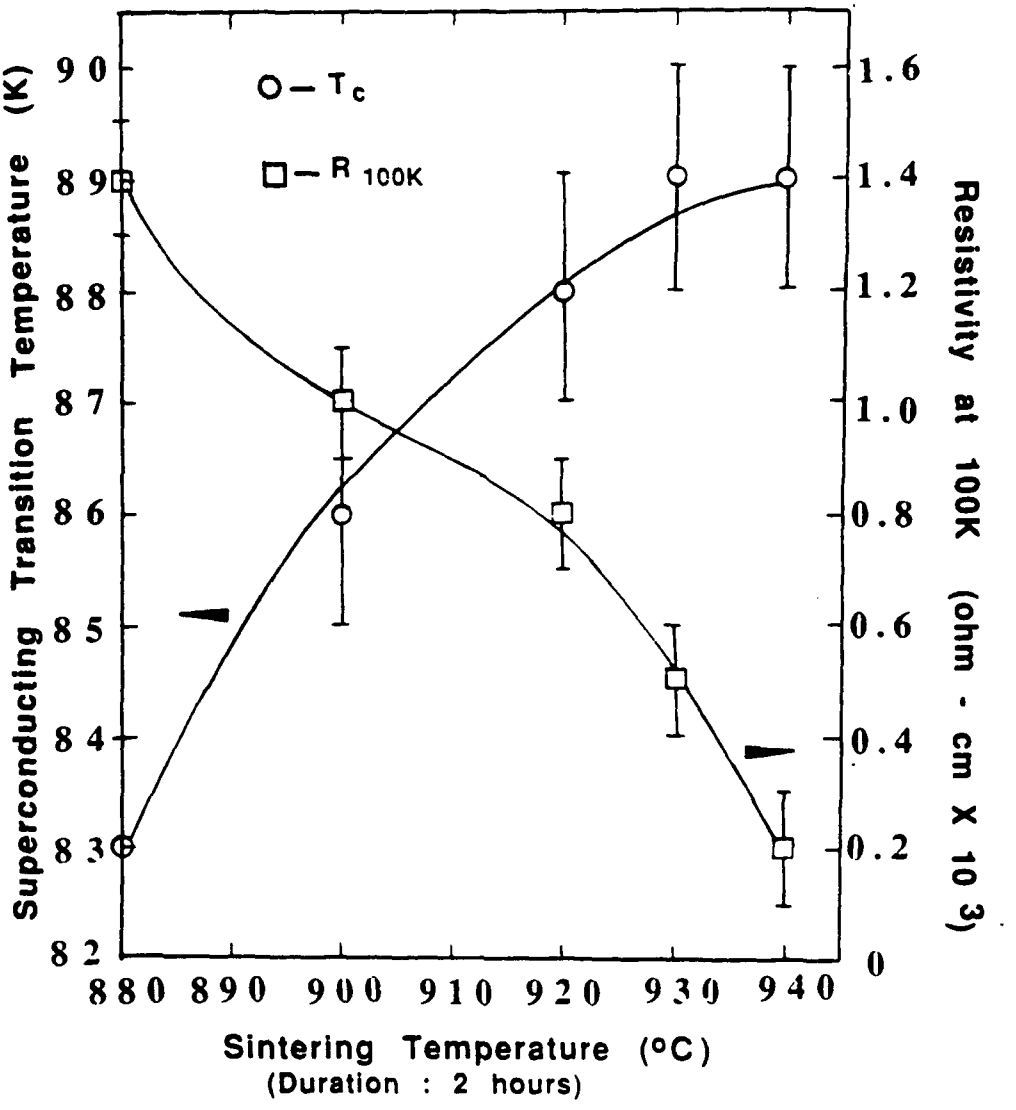

Figure 5. Zero resistance temperature (T_c), and normal state resistivity measured at 100 K (R_{100K}) versus sintering temperature plots of pure $YBa_2Cu_3O_{6+x}$ ceramic materials. Sintering time : 2 hours.

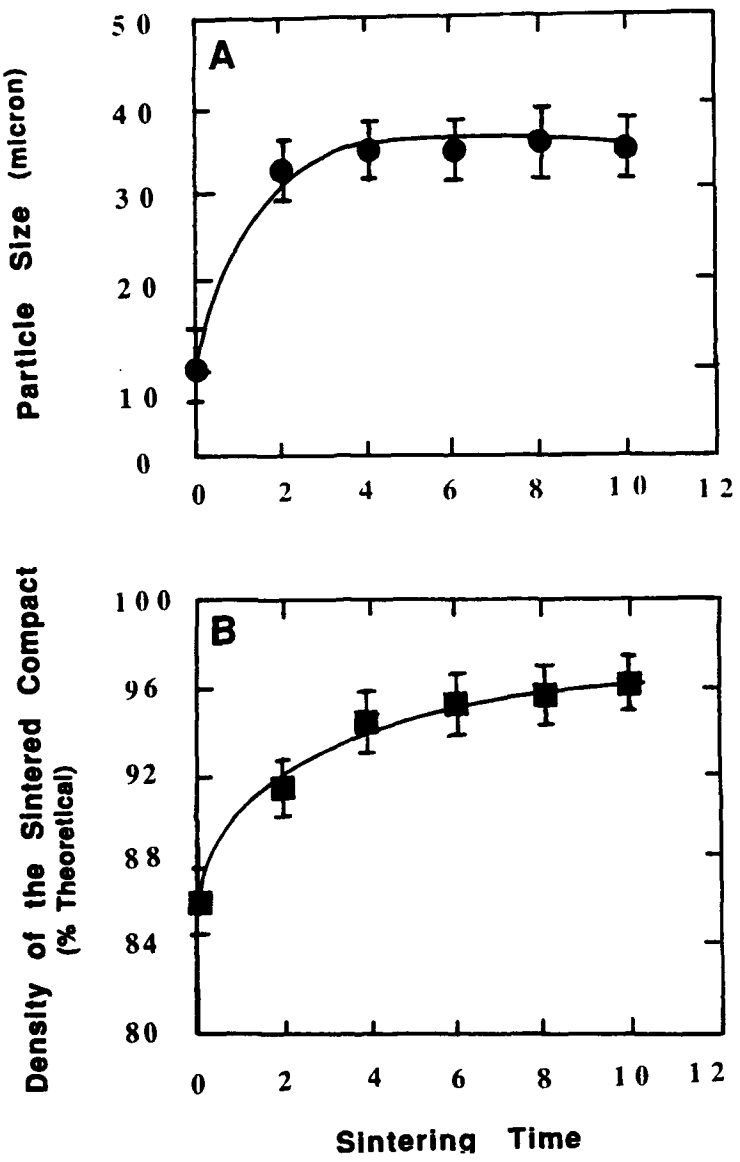

Figure 6. (A) The average particle size of $YBa_2Cu_3O_{6+x}$ and (B) the density of sintered compacts represented as a function of sintering time of pure $YBa_2Cu_3O_{6+x}$ ceramic materials. Sintering temperature 920°C.

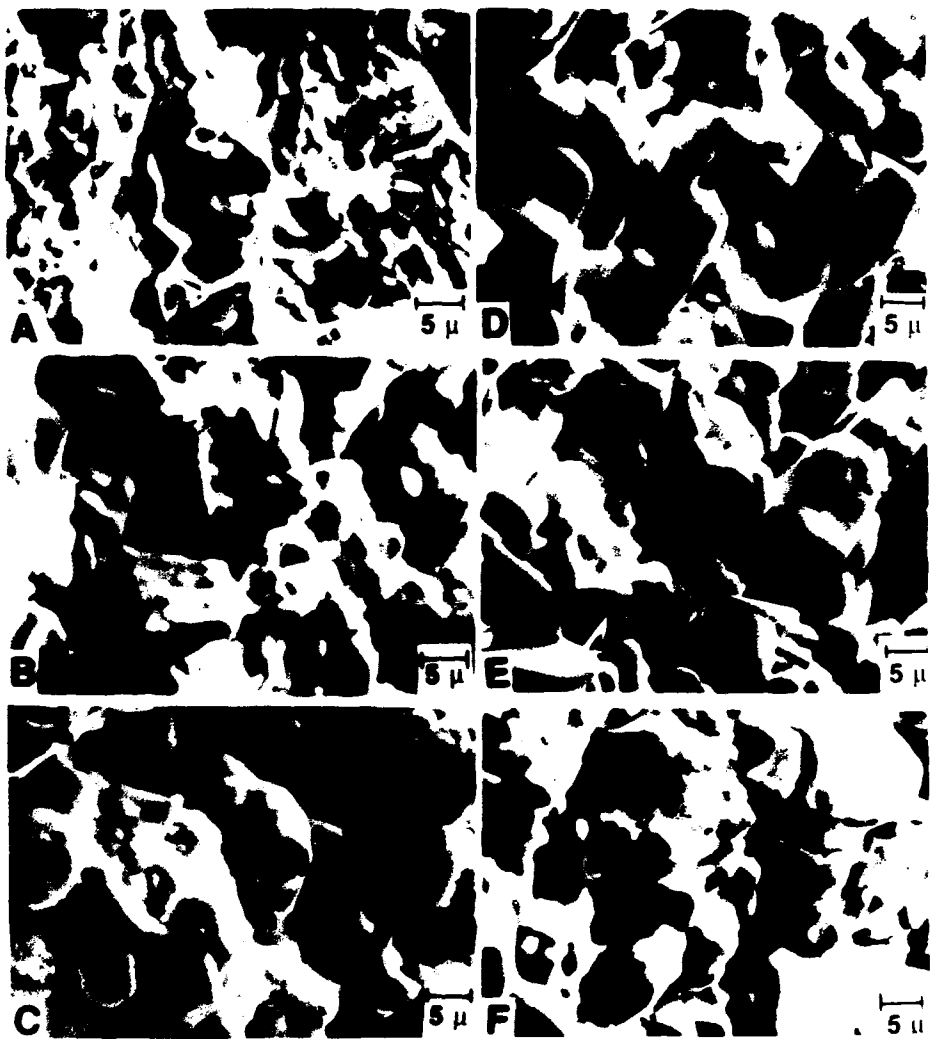

Figure 7. Typical morphology of $YBa_2Cu_3O_{6+x}$ ceramic materials sintered at 920 °C for (A) 0, (B) 2, (C) 4, (D) 6, (E) 8 and (F) 10 hours.

result in a significant melting of the particles. The electrical resistivity measured at 100 K and the zero resistance temperature (T_c) for the above samples is shown in Figure 8 as a function of sintering time. The results suggest that although T_c increases with an increase in the duration of sintering at 920°C during the first 2 hours, prolonged heating (at 920°C) beyond 2 hours does not effect the T_c. The normal state resistivity (measured at 100K) of the sintered samples, on the other hand shows a decrease with an increase in the sintering time over 0 - 6 hour period. Above 6 hours, the duration of sintering has no affect on the sample resistivity.

The average particle size of $YBa_2Cu_3O_{6+x}$ and the sintered compact density of 5 wt.% silver / $YBa_2Cu_3O_{6+x}$ composites sintered for 2 hours and at different sintering temperatures is shown in Figure 9. The results of both the particle size and the bulk density of the silver composites increase with an increase in the sintering temperature. The $YBa_2Cu_3O_{6+x}$ particle morphology in sintered silver/$YBa_2Cu_3O_{6+x}$ composites is shown in Figure 10 as a function of sintering temperature. The results indicate that the surface features such as the particle coarseness decreases with an increase in the sintering temperature. In addition, the results also suggest that silver tends to flow and form unevenly distributed aggregates during sintering for 2 hours above 920°C. A careful examination of the microstructure of the polished surfaces of the sintered composites reveals that the tendency of the $YBa_2Cu_3O_{6+x}$ that are in the vicinity of large concentration of silver particles is to align in the direction of the silver aggregate. Figure 11

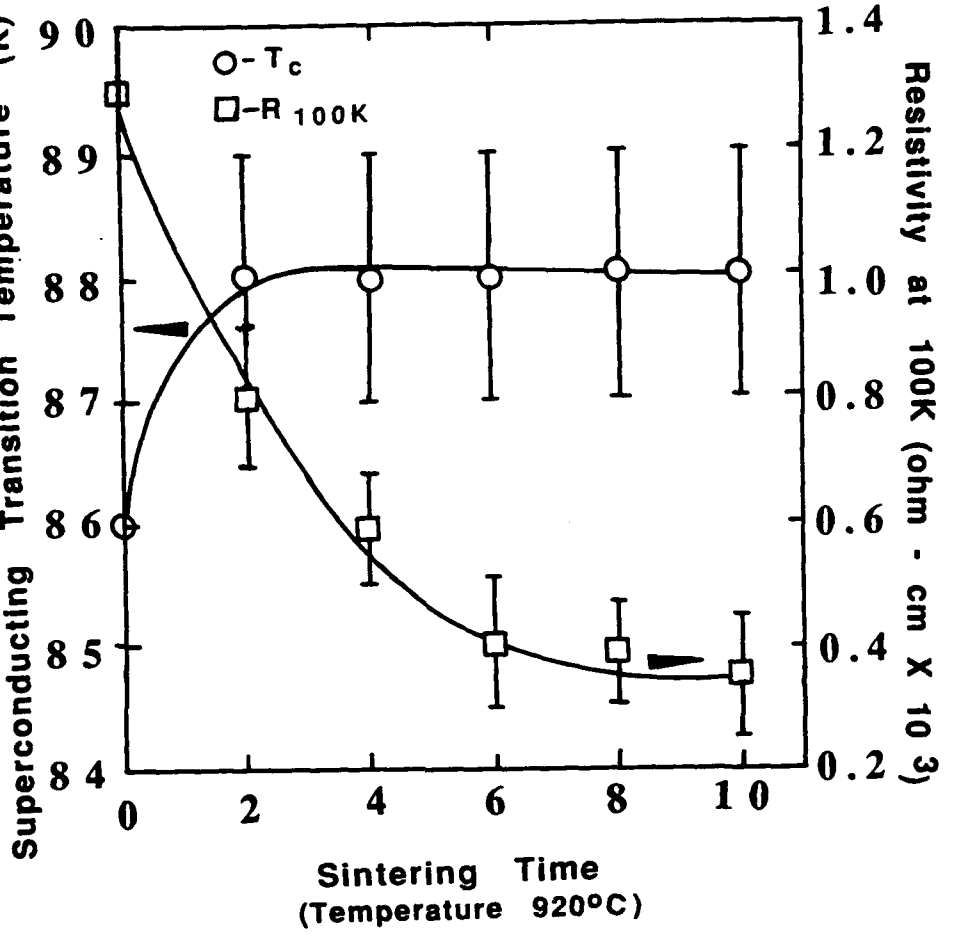

Figure 8. Zero resistance temperature (T_c), and normal state resistivity measured at 100 K (R_{100K}) versus sintering time plots of pure $YBa_2Cu_3O_{6+x}$ ceramic materials sintered at 920°C.

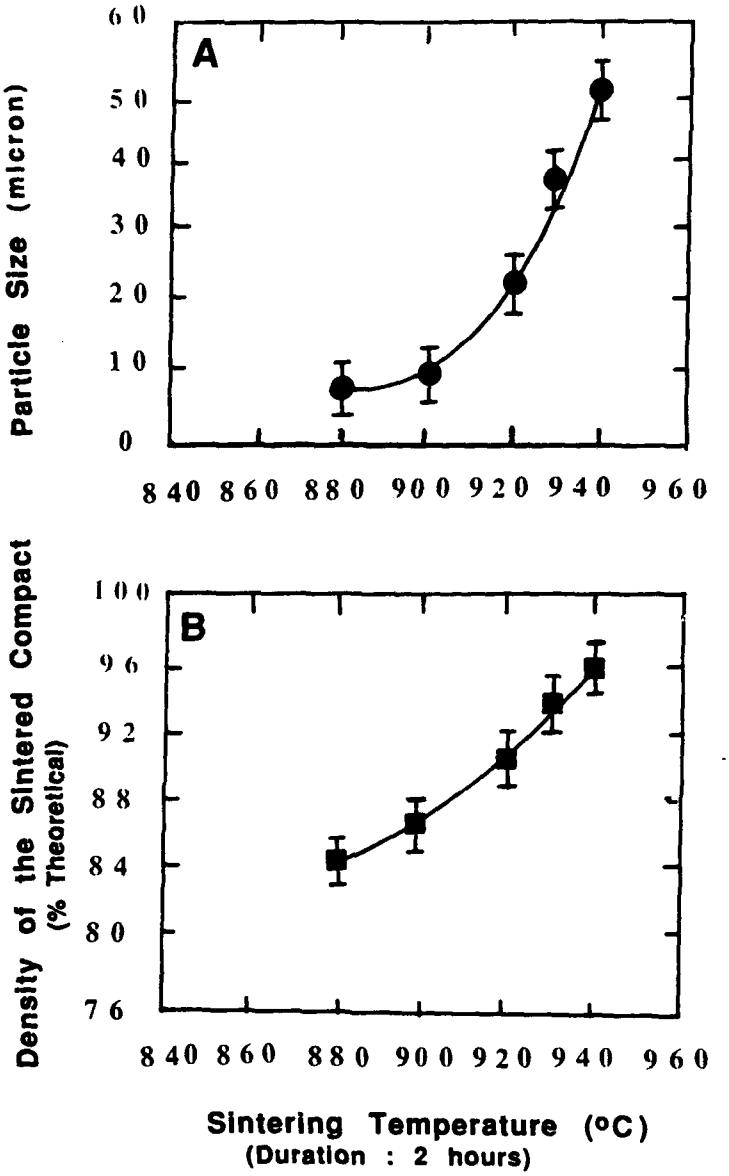

Figure 9. (A) The average particle size of $YBa_2Cu_3O_{6+x}$ and (B) the density of sintered compacts represented as a function of sintering temperature of 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites. Sintering time : 2 hours.

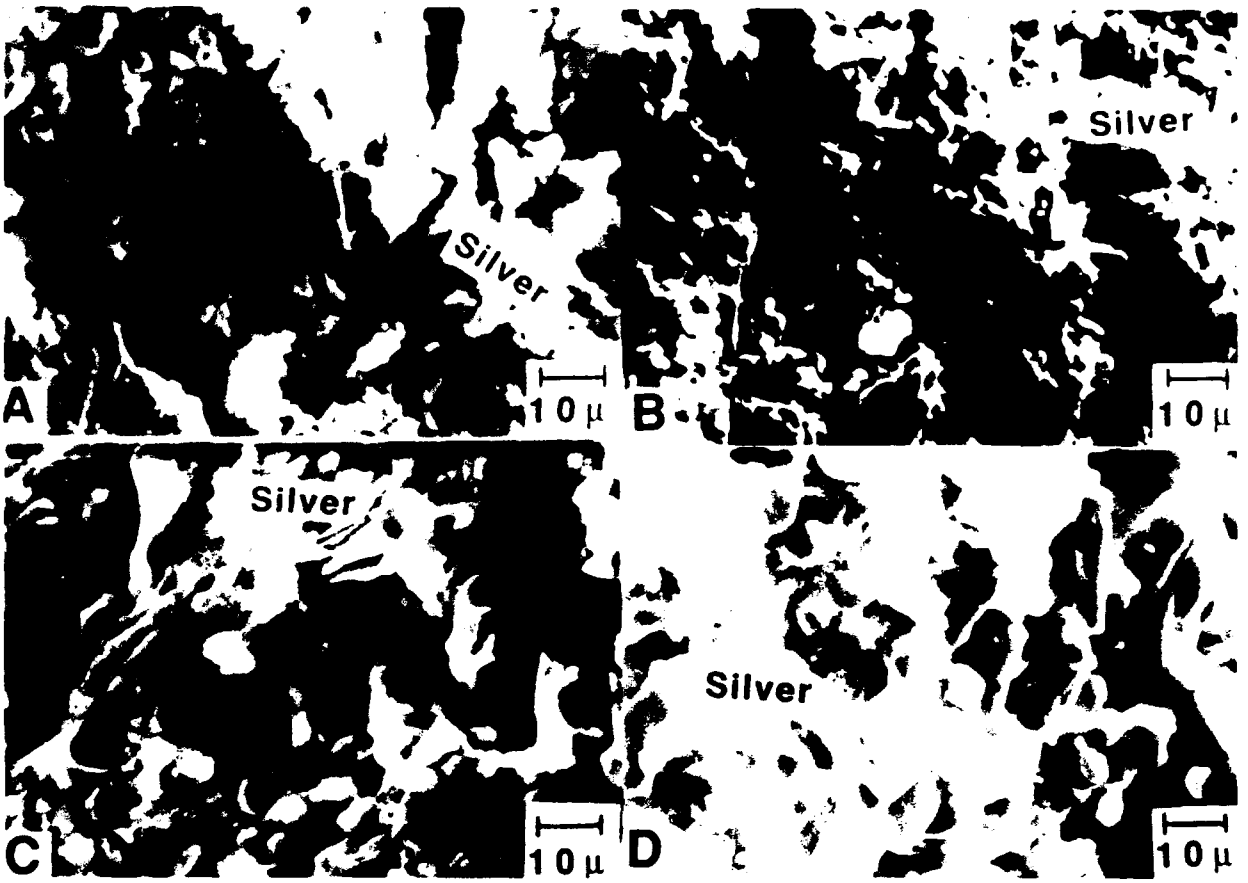

Figure 10. Typical morphology of 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites sintered at (A) 900, (B) 920, (C) 930 and (D) 940°C for 2 hours.

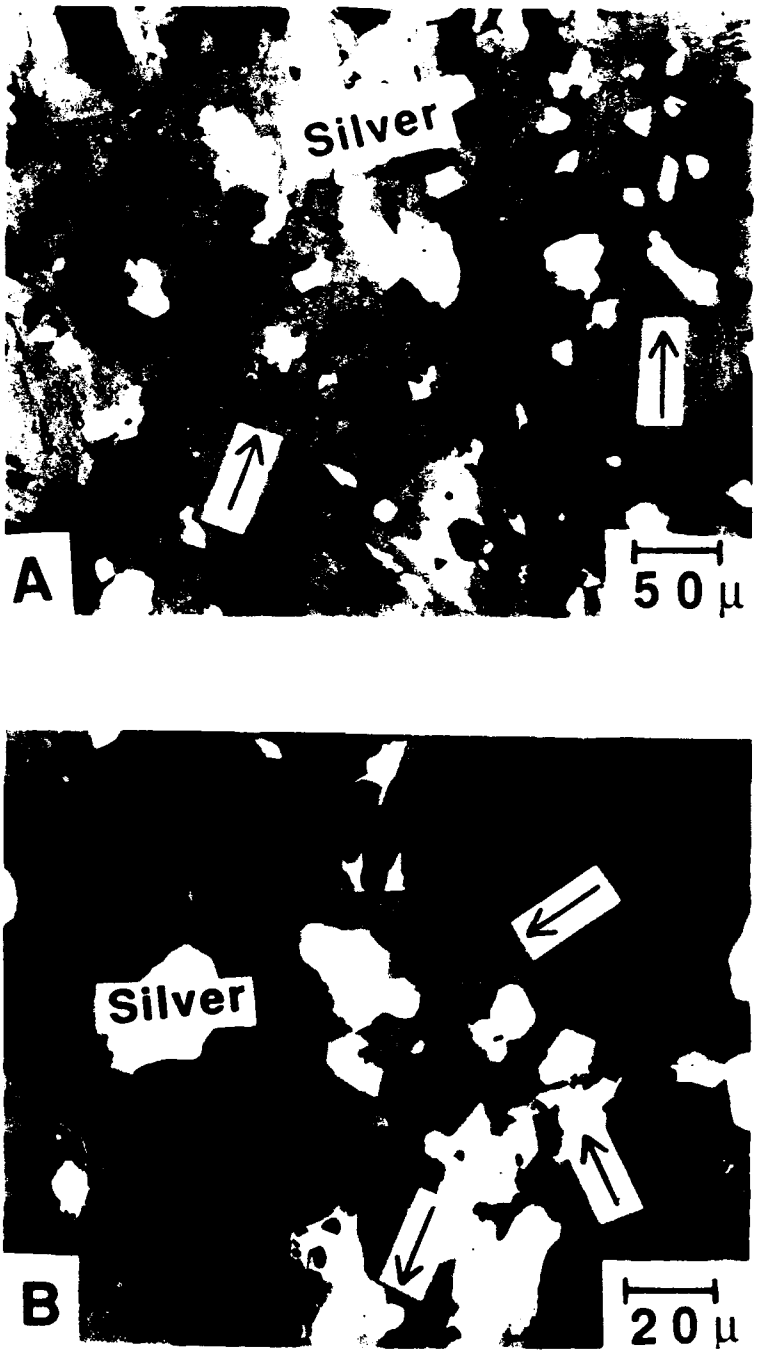

Figure 11. The microstructure of the polished surface of 5 wt.% silver/$YBa_2Cu_3O_{6+X}$ composite sintered at 920°C for 2 hours.

shows a typical microstructure of polished surface of a 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composite sintered at 920°C for 2 hours. A possible mechanism for this grain alignment can be suggested as follows : as the silver/$YBa_2Cu_3O_{6+x}$ composite is heated to the sintering temperature above 920°C, the silver becomes more mobile (because of the fact that the silver is close to it's melting temperature of ~ 960°C) and easily migrates to and fills the voids in the structure. As a result the $YBa_2Cu_3O_{6+x}$ particles are forced to align in the direction of silver flow. This process not only improves the density of the sintered compact (density of the compact sintered at 940°C for 2 hours : pure $YBa_2Cu_3O_{6+x}$ ~ 94% and 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composite ~ 96%) but also it's mechanical integrity. The superconducting zero resistance temperature, and normal state resistivity measured at 100 K versus sintering temperature (sintering time 2 hours) plots of silver composites is shown in Figure 12. The results suggest that while T_c increases, the resistivity of the composites decreases with an increase in the sintering temperature.

The average particle size of $YBa_2Cu_3O_{6+x}$ and density of the sintered composites is shown in Figure 13 for samples sintered at 920°C for 0 - 10 hours. The particle morphology of the above samples is shown in Figure 14. The results suggest that the particle size increases due to calcination at 920°C during the first two hours and remains independent of the sintering time during 2 - 6 hours of calcination. Prolonged calcination above 6 hours appears to re-initiate the $YBa_2Cu_3O_{6+x}$ particle growth. The results on sintered compact density indicates that the composites acquire

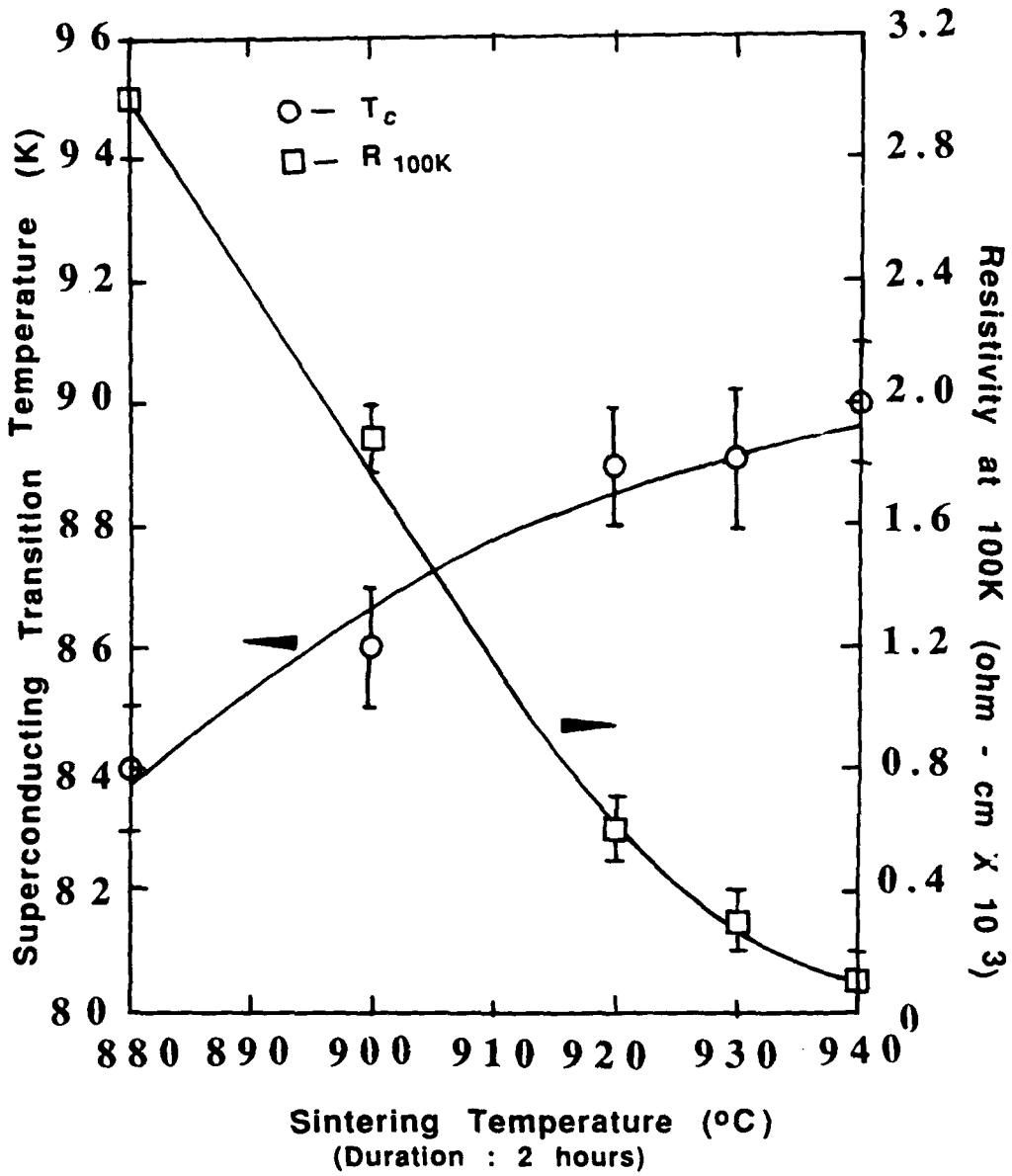

Figure 12. Zero resistance temperature (T_c), and normal state resistivity measured at 100 K (R_{100K}) versus sintering temperature plots of 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites. Sintering time : 2 hours.

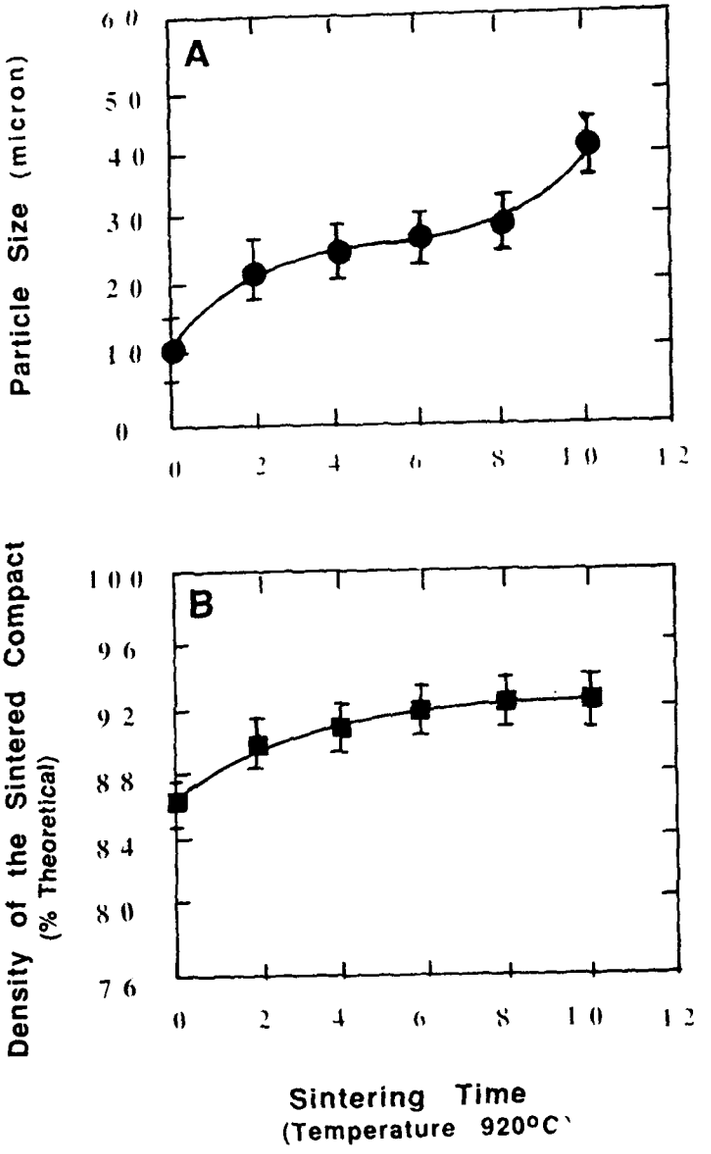

Figure 13. (A) The average particle size of $YBa_2Cu_3O_{6+x}$ and (B) the density of sintered compacts represented as a function of sintering time of 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites. Sintering temperature 920°C.

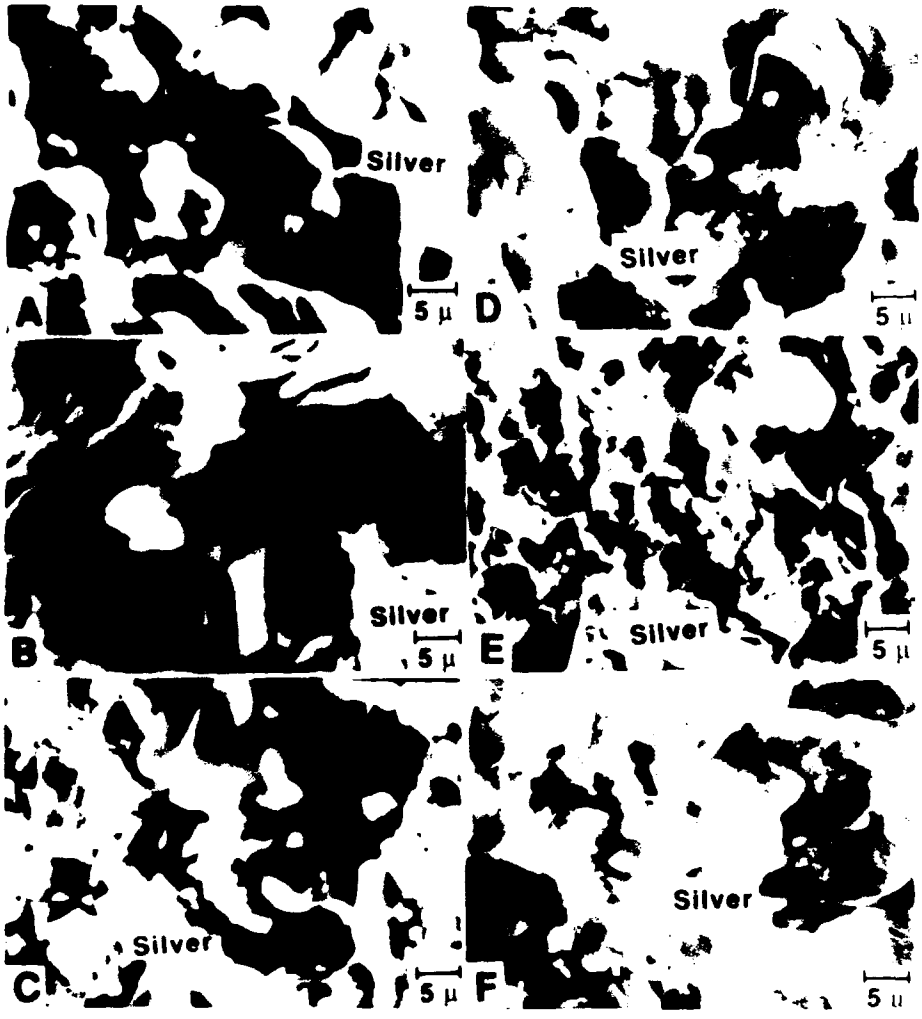

Figure 14. Typical morphology of 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites sintered at 920 °C for (A) 0, (B) 2, (C) 4, (D) 6, (E) 8 and (F) 10 hours.

nearly 90 % of the final compact density during the first 2 hours of calcination at 920°C. Prolonged sintering at 920°C beyond 2 hours does not significantly effect of the density. The significant change in the particle morphology appears to be the development of fine surface topology followed by a partial melting of the $YBa_2Cu_3O_{6+x}$ particle. Prolonged calcination at 920°C tends to increase the melting of the grains, the formation of large clusters of silver and the alignment of the $YBa_2Cu_3O_{6+x}$ particles that are very closely situated to the silver clusters.

Figure 15 shows the zero resistance temperature (T_c), and the normal state resistivity measured at 100 K, (R_{100K}), for 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites sintered at 920°C for 0 - 10 hours. The results suggest that the T_c increases with an increase in the sintering time. The R_{100K} on the other hand decreases with an increase in the sintering time; however, prolonged sintering at 920°C for more than 4 hours has no effect on the sample resistivity.

DISCUSSION

Although the earlier research reports on the deformation behavior of the ceramic superconducting materials at high temperature do not suggest the possibility of any degree of superplastic flow in $YBa_2Cu_3O_{6+x}$ [8,9], based on the theory of superplasticity of materials, it can be suggested that the earlier researchers have not satisfied the critical requirement for superplastic behavior of $YBa_2Cu_3O_{6+x}$. For example, the maximum deformation temperature (600°C) may not be enough to induce any plastic flow. Similarly the high strain rates that were used during deformation are not favorable to the ceramic superconductor.

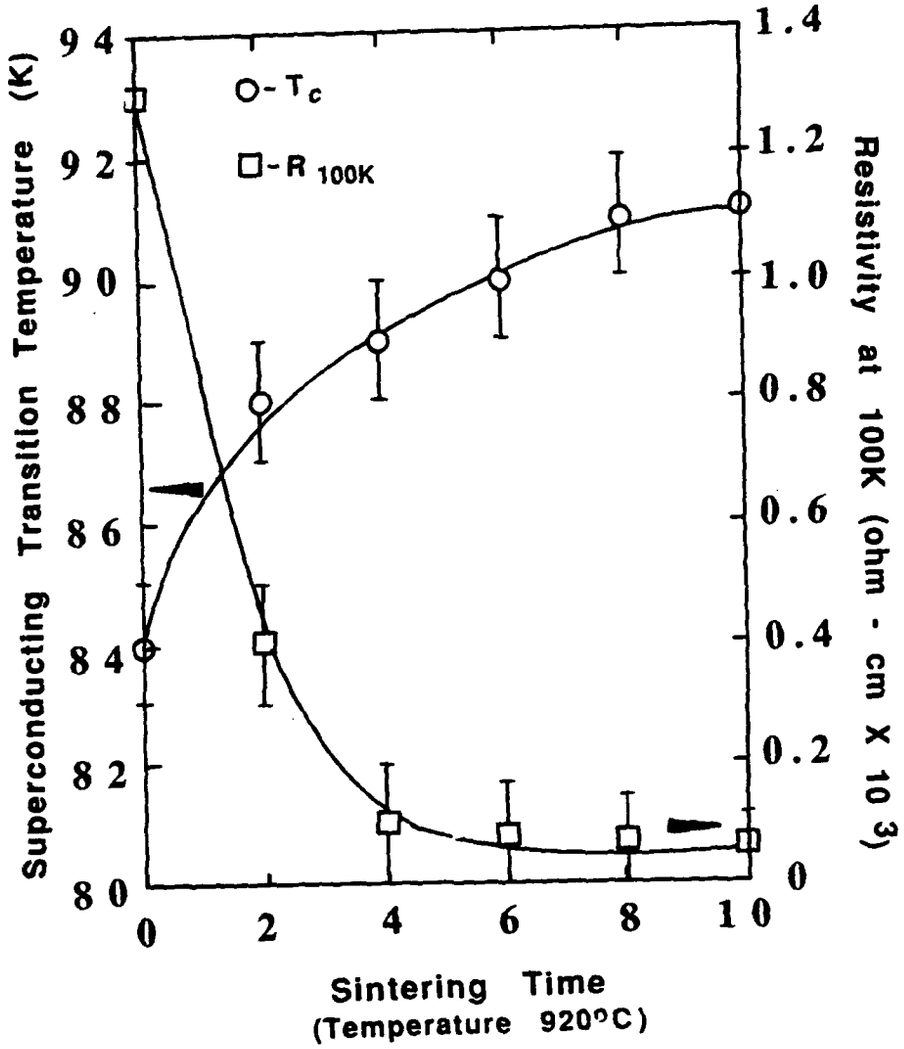

Figure 15. Zero resistance temperature (T_c), and normal state resistivity measured at 100 K (R_{100K}) versus sintering time plots of 5 wt.% silver/$YBa_2Cu_3O_{6+x}$ composites sintered at 920°C.

The important requirements for the superplastic deformation of ceramic materials are : (1) a very small particle or grain size (typically 100 - 200 nm), and (2) the retention of the original crystal structure after deformation. However, the superconducting ceramic materials must also have to satisfy two additional requirements, namely that (3) the superconducting transition temperature (T_c), and (4) the critical current carrying capacity (J_c) have to be unaffected by the deformation process. The present investigation clearly demonstrates that the sintered $YBa_2Cu_3O_{6+x}$ ceramic materials (with and without silver) do not satisfy the first requirement (viz. the small particle size). However, the results of this investigation have provided important information regarding grain growth of $YBa_2Cu_3O_{6+x}$ during sintering for a given set of sintering conditions (i.e. the maximum and minimum value of the $YBa_2Cu_3O_{6+x}$ particle size). In addition the results have suggested that the addition of silver to $YBa_2Cu_3O_{6+x}$ does not reduce the particle size. Therefore for the particle size reduction, the selection of another suitable additive is required.

From the present investigation it is clear that both sintering conditions and the additive have to be changed. If one can model the process of the grain growth (for given set of parameters) of $YBa_2Cu_3O_{6+x}$, it may be possible to predict the required process conditions for obtaining ceramic preform with small particle size. In an attempt to develop suitable conditions for the sintering process, a mathematical analog for the grain growth process using stochastic theory is being developed. The present model assumes

that both kinetic and thermodynamic parameters influence the grain growth process simultaneously.

The preliminary results does not show any correlation between the particle sizes that have been estimated from (i) the mathematical model and (ii) the experimental determination. It has to be pointed out that as a first attempt to model the grain growth process, it was assumed that both the primary particles and the sintered particles were spherical. However, from the microstructural characterization (Figures 3, 7, 10, 11 and 14), it is evident that the $YBa_2Cu_3O_{6+x}$ particles are not spherical, therefore it is expected that our present model would not predict the grain growth of $YBa_2Cu_3O_{6+x}$. At the present time, our mathematical models are being redefined in order to incorporate the effect of shape on grain growth. It is hoped that the new models will enable us to determine optimum sintering parameters for the processing of fine grained $YBa_2Cu_3O_{6+x}$ ceramic preforms that are suitable for superplastic deformation studies.

CONCLUSION

From the present investigation the following conclusions can be derived.

1. The particle size of $YBa_2Cu_3O_{6+x}$ and the sintered compact density increase with an increase of the sintering temperature in the range 880 - 920^{o}C for 2 hours. Above 920^{o}C, the sintering temperature (in the range 920 - 940^{o}C) has no significant effect either on the particle size or the density.

2. The superconducting transition temperature (zero resistance temperature (T_c)) of sintered $YBa_2Cu_3O_{6+x}$ ceramic material increases while the normal state resistivity (measured at 100 K (R_{100K})) decreases due to an increase in the sintering temperature.
3. The surface coarseness of the $YBa_2Cu_3O_{6+x}$ tends to decrease due to an increase in the sintering temperature. If the sintering temperature is $\geq 920^oC$, a partial melting of the $YBa_2Cu_3O_{6+x}$ occurs and the melting process increases with an increase in the sintering temperature.
4. During sintering at 920^oC, the particle size tends to increase with the sintering time during the first two hours of sintering. Prolonged sintering above 2 hours has very little effect on the particle size.
5. The sintered compact density of $YBa_2Cu_3O_{6+x}$ increases with an increase in the sintering time from 0 - 6 hours. Prolonged sintering at 920^oC for more than 6 hours has very little effect on the bulk density.
6. The zero resistance temperature (T_c) increases from ~ 86 K to ~ 88 K during the first 2 hours of sintering at 920^oC. Prolonged sintering above 2 hours has no effect on the T_c. However, the normal state resistivity decreases significantly during the first 6 hours of sintering and remains independent of the sintering time beyond 6 hours.
7. Partial melting of the superconducting $YBa_2Cu_3O_{6+x}$ occurs for sintering at 920^oC for more than 2 hours, and the melting of the particles increases with an increase in the sintering time.

8. An increase in the sintering temperature increases the grain size of $YBa_2Cu_3O_{6+x}$ particles and the sintered compact density in 5 wt.% silver/95 wt.% $YBa_2Cu_3O_{6+x}$ composites sintered for 2 hours.
9. The addition of 5 wt.% silver to the superconducting $YBa_2Cu_3O_{6+x}$ improves the T_c and decreases the normal state resistivity at 100 K. of the material.
10. An increase in the sintering temperature (a) increases the zero resistance temperature T_c and (b) decreases the normal state resistivity measured at 100 K of the composites. The particle size of $YBa_2Cu_3O_{6+x}$ in silver composites increases with an increase in the sintering time at 920°C. However, the sintering at 920°C for 4 to 8 hours does not change in the particle size.
11. During the first four hours of sintering at 920°C, the sintered compact density increases with an increase in the sintering time. Above 4 hours, the increase in the compact density is not very significant.
12. An increase in either the sintering temperature (from 880 to 940°C, and sintering time 2 hours) or the sintering time (sintering temperature 920°C) not only increases the partial melting of $YBa_2Cu_3O_{6+x}$ and the silver particles but also encourages the formation of large clusters of silver filling the voids.
13. The sintering of silver composites above 920°C for 2 hours tend to induce some $YBa_2Cu_3O_{6+x}$ particle alignment.

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The effect of the sintering temperature, the sintering time and the addition of 5 wt. % silver on the average particle size, bulk density and the superconducting zero resistance temperature (T_c) $YBa_2Cu_3O_{6+x}$ ceramic materials was investigated (a) in the range 880 - 940°C for 2 hours and (b) at 920°C for different times in the range 0-10 hours. The results suggest that while the density and particle size of the sintered ceramic increases with an increase of the sintering temperature (and 2 hours of sintering), prolonged sintering (at 920°C) above 4 hours of duration does not improve above properties. However, the superconducting zero resistance temperature (T_c) does not change significantly. In the presence of 5 wt. % silver, it appears that both sintering temperature and duration of sintering significantly improves the bulk density, particle size and (T_c) of $YBa_2Cu_3O_{6+x}$.

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